REMEDIAL ACTION
ROCKWOOL INDUSTRIES, INC. SUPERFUND SITE
BELTON, TEXAS
EPA ID NO. TXD066379645

DEMONSTRATION OF METHODS APPLICABILITY REPORT

Prepared for

U.S. ENVIRONMENTAL PROTECTION AGENCY
1445 Ross Avenue
Dallas, TX 75202-2733

Contract No.	:	68-W6-0037
Work Assignment No.	:	127-RARA-06DJ
Date Prepared	:	June 15, 2005
Prepared By	:	Tetra Tech EM Inc.
Tetra Tech Project Manager	:	Luis Vega
Telephone No.	:	(214) 740-2007
EPA Work Assignment Manager	:	Shawn Ghose
Telephone No.	:	(214) 665-6782
EPA Alternate Work Assignment Manager	:	Gary Miller
Telephone No.	:	(214) 665-8318

CONTENTS

Appendix

A   XRF DATA SUMMARY TABLES
B   DMA STATISICAL DATA TABLES
C   DMA STATISICAL DATA GRAPHS

Attachment

1   TIER IV LABORATORY DATA

CONTENTS (Continued)

FIGURE

ACRONYMS AND ABBREVIATIONS

bgs	Below ground surface
COC	Contaminant of concern
CSP	Cemetery Shot Pile
DMA	Demonstration of methods applicability
DSP	Dangerfield Slag Pile
EPA	U.S. Environmental Protection Agency
EVL	Evaporation Lagoon
FBAL	Field-based action levels
FBDR	Final Basis of Design Report
FPXRF	Field-portable x-ray fluorescence
FSP	Field sampling plan
ICP	Inductively-coupled plasma
LRB	Leon River Bank
mg/kg	Milligram per kilogram
NSP	North Shot Pile
OU	Operable unit
PRG	Preliminary remediation goal
ppm	Part per million
QAPP	Quality assurance project plan
r^2	Coefficient of determination
RA	Remedial action
RI	Remedial investigation
RPD	Relative percent difference
Rockwool	Rockwool Industries, Inc.
RSD	Relative standard deviation
SAP	Sampling and analysis plan
SOP	Standard operating procedure
SSP	South Shot Pile
Tetra Tech	Tetra Tech EM Inc.
VSP	Visual Sampling Plan
XRF	X-ray fluorescence

1.0   INTRODUCTION

Tetra Tech EM Inc. (Tetra Tech) received remedial design Work Assignment No. 125-RDRD-06DJ from the U.S. Environmental Protection Agency (EPA) under Response Action Contract No. 68-W6-0037, on December 15, 2004. On February 11, 2005, Tetra Tech received Work Assignment Form Revision No. 4, which directed Tetra Tech to prepare a field sampling plan (FSP; Tetra Tech 2005a) that, in conjunction with the quality assurance project plan (QAPP; Tetra Tech 2005b), comprises the overall sampling and analysis plan (SAP) for RA field activities at the Rockwool Industries, Inc. (Rockwool) Superfund site in Belton, Texas. Under remedial action (RA) Work Assignment No. 127-RARA-06DJ for the Rockwool site, Tetra Tech conducted a demonstration of methods applicability (DMA) as outlined in the FSP (Tetra Tech 2005a).

The primary purpose of this report is to evaluate an analytical alternative to conducting fixed laboratory analysis of metals in soil and waste materials at the Rockwool site. This alternative will be used to expedite decision-making in the field during excavation activities being conducted as part of this work assignment. As such, a DMA was conducted to determine if the use of field-portable x-ray fluorescence (FPXRF) is a viable alternative to analysis of metals in soils using a fixed-laboratory inductively-coupled plasma (ICP) technique. In addition, this DMA also evaluated two operational modes (described in Section 3.1), two sample preparation techniques (described in Section 3.2), and two instrument set-up modes (count times described in Section 3.3) for the FPXRF instrument to determine the most effective and precise standard operating procedure (SOP) for the analysis. This report (1) summarizes the results of the DMA study conducted from March 28 through 31, 2005, (2) evaluates FPXRF method performance against ICP, and (3) establishes initial field-based action levels (FBAL) and sampling scheme.

The excavation areas shown for the Geer Property-Cemetery Area, North Property, and Operable Unit 2 (OU2) and the Central Property areas are shown on Design Drawings C-5, C-6, and C-8 of CH2M HILL‘s Final Basis of Design Report (FBDR) and Addendum No. 1 (2005a; 2005b). In their FBDR and Addendum No. 1, CH2M HILL divided each excavation area into grid cells and boundary coordinates as shown on the design drawings to define the limits of excavation. Cut depths for each cell were estimated from data collected during the remedial investigation (RI). Cut depths in the Geer Property-Cemetery Area and North Property were limited to a maximum depth of 2 feet because the direct-contact exposure pathway is based on the 0 to

2-foot depth interval. Excavation cells will be field-located by a surveyor using the boundary coordinates provided on the drawings, and the cut depths will be verified by the surveyor following excavation.

In the Central Property, the Dangerfield Slag Pile (DSP) and South Shot Pile (SSP) are enclosed by the "Waste Pile Area" boundary line shown on Design Drawing C-8 (CH2M HILL 2005a; 2005b). Within this area, visible waste above ground surface will be removed, even if concentrations of contaminants of concern (COC) are below direct-contact preliminary remediation goals (PRG). Excavated material with COC concentrations below direct-contact PRGs may be segregated, if feasible, based on FPXRF and verified by Tier IV laboratory analysis, and placed in the Evaporation Lagoon (EVL) with similar low-level waste materials. These areas have also been divided into grids with final grade elevations for each grid cell specified on the drawing. The remainder of the grid cells in the OU2 and Central Property areas will be excavated per the cut depths shown on Design Drawing C-8 (CH2M HILL 2005a; 2005b), and confirmation samples will be collected to determine if additional excavation is required.

2.0   DEMONSTRATION OF METHODS APPLICABILITY STUDY

Prior to initiating the excavation program using x-ray fluorescence (XRF) technology, Tetra Tech conducted a DMA study on several samples representing the range of waste (excavated shot material) and soil (native clay) types across the Rockwool site, which may include the following:

- Visible waste material from waste outcrops located along the Leon River Bank (LRB)

- Waste material from the Geer Property/Cemetery Area

- Waste material from the North Shot Pile (NSP)

- Waste material from the OU2 Property

- Waste material form the Former Baghouse Dust Impoundment Landfill

- Waste material from the SSP (Central Property Area)

- Native clay from beneath the SSP

The DMA study was completed using the FPXRF Niton 700-Series XRF Multi-element Analyzer in conjunction with various sample collection and preparation methods (in situ, ex situ, grinding, drying, and

sieving) for correlation with Tier IV laboratory data. The DMA study evaluated FPXRF performance for all three COCs – antimony, arsenic, and lead.

The following method parameters were evaluated under the DMA:

- Sample analysis operational mode (in situ versus ex situ)

- Various sample preparation techniques and their impacts on observed precision of results relative to decision-making (i.e., the utility of in situ FPXRF measurements versus ex situ FPXRF measurements)

- Instrument count times and analytical measurement conditions and their impact on precision, bias, and decision-making

- Correlation of XRF results using various preparation techniques with Tier IV laboratory analyses using EPA SW-846 Methods 3051/6010B (1996) for site COCs

- XRF sample population distribution, summary statistics, and statistical plots

- Statistical evaluation of the number of samples required to verify that cleanup standards have been met

After conducting sampling activities under the DMA, a computer software program called Visual Sample Plan (VSP; http://dqo.pnl.gov/VSP/) (Pacific Northwest National Laboratory 2004) will be used to evaluate how many samples from the remaining facility area are necessary to confirm that COC contamination in waste and soil exceeding cleanup goals are detected with a specified level of confidence and significance. VSP uses inputs for the standard deviation of a specific population, a specified level of confidence and significance, and the total number of samples anticipated to be collected to identify a region of uncertainty or safety factor beneath an action level from a decision-making standpoint. That is, the concentration beneath a specific action level where a decision cannot be made within the required level of confidence unless additional data is gathered.

The method and the FBAL can (and should) continue to be adapted and refined as the project proceeds. For example, VSP and other decision support tools can be used to assess whether the samples from different properties are fundamentally different populations. Numbers of samples needed and FBALs can then be refined as necessary for the decision needs at each property. The FPXRF FBAL will be refined throughout the RA.

3.0   SAMPLE COLLECTION, PREPARATION, AND ANALYICAL PROCEDURES

Two instrument operational modes, two sample preparation methods, and two instrument settings were evaluated to determine which is the most effective in obtaining precise analytical results using the FPXRF. The two operational modes include in situ and ex situ (Section 3.1). The two sample preparation techniques include (1) homogenization of the soil matrix only; and (2) drying, particle size reduction, and homogenization of the soil matrix (both discussed in Section 3.2). The two instrument settings included a 60-second and 120-second count time for the instrument measurement (Section 3.3).

3.1   IN SITU VERSUS EX SITU ANALYSIS

In situ (in place) FPXRF analysis is very useful for obtaining decision-making data with minimal sample preparation. In the in situ mode of operation, the analyzer is placed in direct contact with the soil surface to be tested; within seconds, a value of ± 50 to 60 parts per million (ppm) lead (for example) is displayed for a low-resolution standard (www.niton.com).

In situ FPXRF analysis was initially proposed by placing the instrument window directly onto the soil at the base of the excavation or in the excavator bucket as part of this DMA. However, due to rain events and standing water, in situ analysis was not feasible because the excess moisture in the sample matrix might cause analytical interference and possibly generate inaccurate and imprecise data. As such, the ex situ mode was the only mode of operation evaluated during this DMA.

The ex situ FPXRF mode (prepared samples) is more involved than the in situ mode, requiring that a sample be collected and homogenized prior to analysis. This sample preparation technique was selected as the most feasible for obtaining reliable FPXRF results. Section 3.2 describes the two homogenization techniques used for ex situ analysis mode in this DMA.

3.2   HOMOGENIZATION ONLY VERSUS DRYING AND HOMOGENIZATION

For analyzing samples in the ex situ mode, samples must be collected from this site location and prepared prior to analysis. Two sample preparation procedures were evaluated: (1) minimal homogenization that included manual removal of rocks, organic matter, and large soil grains followed by kneading of the soil
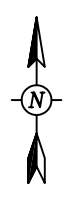
material to enhance mixing; and (2) rigorous homogenization that involved oven drying and particle size reduction, followed by blending. Both methods were applied during the DMA, and each was compared with Tier IV laboratory results for split samples to determine which method would be used for the remainder of the RA.

For the purposes of this DMA, soil samples for ex situ XRF and Tier IV laboratory analysis were collected directly from the excavator bucket or the base of the excavation using dedicated sampling equipment (stainless-steel scoop or spoon), and placed into a resealable plastic bag. Soil samples were collected from 10 locations throughout the site including the Geer Property/Cemetery Area, LRB, and the Central Property, which includes the former Bag House Dust area landfill, SSP, and DSP; see Figure 1 for sampling locations. Several additional samples, which were collected during the DMA study but not included as part of the DMA, are also indicated on Figure 1.

The soil samples were collected from various depths. The following is a list of the sample identifiers and their collection depths:

- DMA-LRB-A-01 was collected from the surface of the southwestern bank of the Leon River (LRB)
- DMA-CSP-D2-01 was collected from 2 feet below ground surface (bgs) at the Cemetery Shot Pile (CSP)
- DMA-NSP-C3-01 was collected from 2 feet bgs at the NSP
- DMA-CP-J7-01 was collected from 3.5 feet bgs in the Central Property
- DMA-BHD-I3-01 was collected from 8 feet bgs in the former BHD area landfill
- DMA-BHD-I3-02 was collected from 4 feet bgs in the former BHD area landfill
- DMA-SSP-D7-01 was collected from 5 feet bgs in the SSP
- DMA-SSP-E8-03 was collected from 7 feet bgs in the SSP
- DMA-SSP-C9-04 was collected from 7 feet bgs in the SSP
- DMA-SSP-C9-05 was collected from the surface of the SSP

One duplicate sample (DMA-LRB-A-01-99) was collected from the LRB to measure the influence of sampling and field procedures on the precision of the environmental measurement. Field duplicate samples are independent samples collected as close as possible, in space and time, to the original sample.

ES
6

CALLED 7.29 ACRES
O'DELL GEER
VOL. 4123 PG. 746
D.R.B.C.T.

⊗ DMA-LRB-A-01

LEON RIVER

⊗ DMA-CSP-D2-01

CEMETERY

CEMETERY

NORTH SHOT PILE

⊗ DMA-NSP-C3-01

EVAPORATION
LAGOON

FRESHWATER
PROCESS BLDG

OFFICE
BUILDING

ASPHALT

EAST 9th AVENUE

HEADWALL

ASPHALT PAVEMENT

WATER
CONCRETE

⊗ SAMPLE
PIT-GT-3

Baghouse Dust
Wastepond Class 1 TXU GAS
LANDFILL (CLOSED)

⊗ DMA-BHD-I3-02
⊗ DMA-BHD-13-01

LSG (GAS)

M. K. & T. RAILROAD R.O.W.

⊗ SAMPLE
PIT-GT-2

WAREHOUSE
BUILDING

⊗ DMA-CP-J7-01

WATER

⊗ SAMPLE
PIT-GT-01

4 BOLLARDS

BRICK
PLANT

DANGERFIELD
SLAG PILE

MAINTENANCE
BUILDING

WASTEWATER
BLENDING TANK

⊗ DSP-E3-01

STORMWATER
RUNOFF POND CLASS 1
LANDFILL (CLOSED)

⊗ DMA-SSP-D7-01

⊗ DMA-SSP-E8-03

12" CMP

SOUTH SHOT PILE

⊗ SSP-D10-06
SSP-D10-02
⊗ DMA-SSP-C9-05
⊗ DMA-SSP-C9-04

A 18" RCP

SUSANNE H. BOND
VOL. 1637, PG. 83
D.R.B.C.T.

BARB WIRE FENCE

FM 93

LEGEND

⊗ LRB-A-01   SAMPLE LOCATION
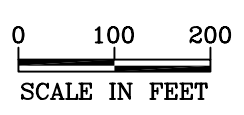
ROCKWOOL INDUSTRIES, INC.
BELTON, TEXAS

FIGURE 1
DMA SAMPLE LOCATIONS

0   100   200

SCALE IN FEET

PREPARED FOR: EPA   BY: Tetra Tech EM Inc.

T:\CADD\G\00DA\1127\DWGs\ SampleLoc01.dwg   05/27/2005 schwerj TX

The samples were homogenized in an aluminum pan or the resealable plastic bag. In order to evaluate the two sample preparation methods, homogenization only and drying plus homogenization, one-half of the sample was stored in a resealable bag for analysis by FPXRF. Results for samples prepared in this manner (homogenized only) are presented in Table A-1 (Appendix A). The remaining one-half of the sample was placed in an aluminum pan, and placed in an oven for a minimum of 4 hours at 104°C. After removing dried samples, they were broken up (if necessary) and homogenized as much as possible. Sieving was not warranted because the data will not be used for human or ecological risk assessment purposes. The dried homogenized samples were placed into a new resealable bag and analyzed using the FPXRF with two instrument settings (count times are discussed in Section 3.3). Results for samples prepared by this method (drying plus homogenization) are also summarized in Table A-1 (Appendix A).

To verify the precision of the FPXRF analyses, two of the 10 samples collected as part of the DMA were selected for replicate measurements. Samples DMA-SSP-C9-05 and DMA-BHD-I3-01 were chosen for replicate measurements because all three COCs were detected above their respective PRGs. Ten replicate measurements were obtained for each sample, and the percent relative standard deviation (RSD) was used to validate the FPXRF results. The precision is acceptable because the percent RSD for the replicate measurements was less than 30 percent for each analyte. Results of the replicate measurements and calculated relative percent differences (RPD) are included in Tables A-2 and A-3 (Appendix A) for DMA-SSP-C9-05 and DMA-BHD-I3-01, respectively.

The evaluation of data collected to determine the most feasible and precise sample preparation technique is discussed in Section 4.1.

3.3   60-SECOND VERSUS 120-SECOND ANALYTICAL COUNT TIME

In order to obtain the most precise measurements on the FPXRF, two count time settings were used to analyze samples prepared by both methods discussed in Section 3.2. Generally, a longer count time produces data with better precision. Two practical count times were selected for evaluation (60 seconds and 120 seconds) at the manufacturer's recommendation. Samples prepared by both methods discussed in Section 3.2 were analyzed at each of the two instrument settings. Results for soil samples are recorded in Table A-1, and results for the 10 replicate measurements are presented in Tables A-2 and A-3 (Appendix A).

Evaluation of data collected to determine the most feasible and precise instrument setting are discussed in Section 4.1.

4.0   STATISTICAL DATA EVALUATION

Three evaluations were conducted on the FPXRF and ICP data obtained from this DMA. The first statistical data evaluation determined the variation associated with different sample preparation techniques and instrument settings (count times). The second statistical evaluation determined the correlation between various FPXRF data sets and fixed-laboratory ICP analyses. The third statistical evaluation determined the data distributions for each of the COCs and whether the data populations were normally or lognormally distributed.

4.1   SAMPLE PREPARATION AND COUNT TIME EVALUATION

This section includes evaluations of homogenized only (non-dried) sample data, dried-homogenized sample data, and the effect of count times on the results. Table B-1 (Appendix B) provides summary statistics for the 60- and 120-second count data obtained from the analysis of homogenized (non-dried) samples. Standard deviation and variance were evaluated to determine which count time produced the best precision (i.e., lower standard deviation and variation). For lead, the standard deviation and variance were slightly lower for the 120-second count time measurements. However, for arsenic and antimony, the standard deviation and variance were lower in the 60-second count time measurements. In homogenized (non-dried) samples, this indicates that the 60-second count time is sufficient because the precision for two (arsenic and antimony) out of three analytes was better, and the precision for lead was acceptable.

The same evaluation was conducted for data obtained from the analysis of dried-homogenized samples. Table B-2 (Appendix B) provides the 60- and 120-second count time DMA data sets for the dried-homogenized samples. This data set indicates significant improvements in standard deviation and variance for arsenic and antimony using the 120-second count times, while the standard deviation and variance values actually slightly increased for lead. These results are inconsistent with the previous evaluation of homogenized only samples and count time discussed in the paragraph above.

As such, neither of the data sets for the homogenized and the dried-homogenized samples definitively indicated which count time produced the most precise data. Therefore, two additional evaluations were conducted. First, the data from the 60- and 120-count time measurements were evaluated against the two preparation methods. Statistics for this evaluation are included in Table B-3 (Appendix B). Combining the count time measurements and evaluating against the two preparation methods produced a consistent result that indicates that homogenization only (non-dried) produced the lowest variability (based on standard deviation and variance).

Second, the homogenized only (non-dried) and dried-homogenized data sets were evaluated against the count time to determine the effects of count times on the standard deviation and variance of the data set. Statistics for this evaluation are included in Table B-4 (Appendix B). This evaluation indicates that the standard deviation and variance were slightly higher for lead at the 120-second count time, but significantly lower for arsenic and antimony at the 120-second count time. Based on this evaluation, it can be concluded that the 120-second count time would significantly decrease the standard deviation and variance and, thereby, increase the precision of the measurement.

Combining the conclusions of these evaluations, it appears that the most precise data were obtained by using the homogenization only (non-dried) sample preparation method and the longer (120-second) count time.

4.2   CORRELATIONS BETWEEN DATA SETS AND FIXED LABORATORY ANALYSES

All 20 samples plus two duplicate samples were submitted for Tier IV laboratory analysis using EPA SW-846 Method 6010B (ICP) (1996) for antimony, arsenic, and lead. Attachment 1 presents the Tier IV laboratory data. The evaluation of sample preparation and comparison to laboratory ICP results are discussed in Section 4.0.

The summary statistics discussed in Section 4.1 provided a good foundation; however, the differences in standard deviation and variance were not consistently significant based on changes to sample preparation or count times. While a general conclusion was drawn, the summary statistics were often inconsistent for each of the COCs, which indicated that increased count times or sample preparation was beneficial for certain metals but not others. Based on these indications, it was considered necessary to develop

correlations for various sample preparation and count times versus fixed laboratory analyses to provide an indication of which FPXRF SOP would provide the best correlation with collaborative ICP results. Two evaluations were conducted: (1) FPXRF data obtained from homogenized only (non-dried) samples for each of the two count times (60 and 120 seconds) were evaluated against ICP results; and (2) FPXRF data obtained from the 120-second count time for each of the two preparation methods were evaluated against ICP results.

In the first evaluation, linear regression curves were plotted and correlations were developed for each of the COCs using homogenized FPXRF results for the 60- and 120-second count times versus corresponding concentrations from the Tier IV laboratory ICP SW-846 Method 6010 analysis (EPA 1996). Graph C-1 (Appendix C) provides the linear regression plots and correlations between the 60- and 120-second homogenized samples versus fixed laboratory results for each of the COCs. The calculated correlation coefficient (r^2) is shown on each plot. It appears that increasing the count times from 60 seconds to 120 seconds produces data with a better correlation for all three COCs. The r^2 increased from 0.9592 to 0.9668 for lead, from 0.7542 to 0.8707 for arsenic, and from 0.9118 to 0.9417 for antimony. The increased r^2 values indicated that the 120-second count time provided significant improvements to correlations with ICP concentrations.

In the second evaluation, linear regression curves were plotted and correlations were developed for each of the COCs using the 120-second count time for each of the preparation techniques versus corresponding ICP concentrations. Graph C-2 (Appendix C) provides the developed correlations for the 120-second homogenized (non-dried) and dried-homogenized data sets versus ICP concentrations for each COC. Additional sample preparation associated with the dried-homogenized data set included (1) drying the sample for at least 4 hours at 104°C and (2) homogenizing the sample prior to analysis. The r^2 value for lead was better for the homogenized only (non-dried) sample preparation, while the r^2 values for arsenic and antimony were better for the dried-homogenized sample preparation. This additional sample preparation resulted in a decreased r^2 value from 0.9668 to 0.9476 for lead, an increased r^2 value from 0.8707 to 0.8848 for arsenic, and an increased r^2 value from 0.9417 to 0.9783 for antimony. The inconsistent results of the additional sample preparation and the relatively small change in r^2 values indicate that the time and expense of the additional sample preparation does not provide significant enough improvements in correlations with the ICP data to warrant its use over the simpler homogenization only (non-dried) sample preparation technique.

Combining the conclusions of these evaluations, it appears that the sufficiently precise data were obtained by using the homogenization only (non-dried) sample preparation method and the longer (120-second) count time. This conclusion is consistent with the conclusions derived from the evaluations in Section 4.1.

4.3   EVALUATION OF DATA DISTRIBUTIONS

The third statistical evaluation determined the range of the data set, presence of outliers, and whether the data were normally or log-normally distributed. Based on the conclusions of the two previous evaluations (discussed in Sections 4.1 and 4.2), the dataset used in this evaluation included data obtained from the 120-count time FPXRF analysis of homogenized only (non-dried) samples. The data were evaluated as is (normal) and as log-transformed data. Graphs C-3, C-4, and C-5 (Appendix C) provide box and whisker plots, histograms, and probability plots for each of the 120-second count homogenized (non-dried) data sets for lead, arsenic, and antimony, respectively. Box and whisker plots were used to indicate the range and presence of outliers for both the normal and lognormal data sets. No outliers were present in the box and whisker plots. The histograms, which are used to evaluate how well a particular data set behaves as a normal or lognormal distribution, are of particular importance in these graphs. The Shapiro-Wilk's Test was used to evaluate whether the data set will follow a normal or lognormal distribution. A data set with a Shapiro-Wilk's "p" value greater than 0.05 indicates the distribution is lognormal.

For lead (Graph C-3, Appendix C), the Shapiro-Wilk's "p" value for the untransformed data set is 0.03068, while the "p" value for the log-transformed data set is 0.16530. Because the "p" value for the log-transformed data set (0.16530) for lead was higher than the untransformed data set and the value exceeded 0.05, the lead data have a lognormal distribution. The probability plots indicate that the log-transformed data set follow a linear best fit rather than the untransformed data set for the lead 120-second count homogenized XRF results. Based on the plots provided in Graph C-3 (Appendix C), the lead data distribution is considered lognormal.

The same plots were also developed for the arsenic 120-second count homogenized FPXRF data set (Graph C-4, Appendix C). The box and whiskers plot indicates the range of the dataset and shows no apparent outliers. Unlike lead, the arsenic appears to follow a normal distribution (see probability plots). The Shapiro-Wilk's "p" value was higher for the arsenic untransformed data set (0.08464) than it was for

the log-transformed data set (0.01869) and exceeded the 0.05 criterion. The probability plots also visually show that the untransformed data set follow a linear best fit better than the log-transformed data set for the arsenic 120-second count homogenized FPXRF results. The plots provided in Graph C-4 (Appendix C) indicate that the arsenic distribution is normal.

Finally, the same plots were also developed for the antimony 120-second count homogenized FPXRF data set (Graph C-5, Appendix C). The box and whiskers plot indicates the range of the dataset and shows no apparent outliers. Similar to lead, the antimony distribution visually follows a lognormal distribution (see probability plots), and the Shapiro-Wilk's "p" value was higher for the log-transformed data set (0.28394) than for the untransformed data set (0.00025). Because the "p" value for the log-transformed data set (0.16530) for antimony was higher than the untransformed data set and the value exceeded 0.05, the antimony data have a lognormal distribution. The probability plots also visually show that the log-transformed data set follow a linear best fit better than the untransformed data set for the antimony 120-second count homogenized FPXRF results. Using the plots provided in Graph C-5 (Appendix C), the antimony distribution is considered lognormal.

5.0   FIELD-BASED ACTION LEVELS

Using the preparation and analytical techniques decided in Section 3.0 and the statistics obtained in Section 4.0, FBAL were determined so that near-real-time decisions can be made using the FPXRF data without having to wait on costly and time-consuming fixed-laboratory analysis of metals in soils.

5.1   VISUAL SAMPLING PLAN

VSP was used to determine the number of samples to evaluate and the FBAL for each COC. VSP can determine how many samples from the remaining facility area should be analyzed to confirm that COC contamination in waste and soil exceeding cleanup goals are identified with a specific level of confidence and significance. The information obtained in Section 3.0 was used to create performance curves in VSP, which was used to develop XRF FBAL for each COC. VSP uses inputs for the standard deviation of a specific population, a specified level of confidence and significance, and the total number of samples anticipated to be collected to identify a region of uncertainty or safety factor beneath an action level from

a decision-making standpoint; that is, the concentration beneath a specific action level where a decision cannot be made with the required level of confidence unless more data are gathered.

The values for lead, arsenic, and antimony were input into VSP. VSP requires a null hypothesis, a false rejection rate (alpha), a false acceptance rate (beta), the width of the region of uncertainty (delta), an action level, and a sample population standard deviation.

The null hypothesis was set as "the true median or mean is = to the action level," which means the site is assumed to be contaminated and must be proven to be non-contaminated. This is a conservative null hypothesis that is typically used for environmental cleanup actions. Next, decision errors (alpha and beta) were entered into the VSP input for each performance curve. The decision errors were set at 5 percent for alpha (false rejection) and 10 percent for beta (false acceptance).

The alpha rate was set at a conservative rate (5 percent) because this type of error would result in considering a grid non-contaminated when it is actually contaminated. In other words, the analyte's mean concentration is considered to be below the action level while the actual mean exceeds the action level. The false rejection (Type I error), which is considered more serious than the Type II error (see below), would result in potentially exposing receptors to concentrations above the action level. As a result, the lower 5 percent error tolerance is used.

The beta rate was set at 10 percent because this type of error would result in considering a grid contaminated when it is actually not contaminated. This is to say, the analyte's mean concentration is considered to be above the action level, while the actual mean is lower than the action level. The false acceptance (Type II error), which is considered less serious of the two error types, would result in potentially removing material that does not exceed the action level. Thus, the higher 10 percent error tolerance is used.

The next step was to determine the width of the region of uncertainty for each of the COCs (the uncertainty of each COC is discussed in the respective paragraphs below). The width of the region of uncertainty for the COCs was determined based on the approximate number of samples expected to be collected during the site cleanup activities. There are 72 grids on site requiring characterization and

potential remediation, and each grid will be subdivided into nine sub-grids. Using this logic, a minimum of 648 homogenized samples analyzed for the 120-second count are expected to be collected.

Lastly, VSP developed a performance curve for each COC, which requires an action level and estimated standard deviation. Table B-1 (Appendix B) includes all of the necessary information for entry into VSP for each COC. Performance curves were developed using either the Wilcoxon Signed Rank (One-Sample) Test, which does not require a normal distribution, or the One-Sample t-Test of the True Mean versus the Action Level, which does require a normal distribution.

Lead

The region of uncertainty for lead was developed and set at 0.25 (a log transformed value), which resulted in a minimum required sample population of 630 samples. The region of uncertainty for lead only requires 630 samples; therefore, based on preliminary estimates (648 samples), the region of uncertainty is conservative.

The statistical plots and the Shapiro-Wilk's test (Graph C-3, Appendix C) indicate that the 120-second count homogenized FPXRF lead data set followed a lognormal distribution, and log-transformed values were used for VSP input. The log-transformed value of the lead action level (1,754 milligrams per kilogram [mg/kg]) is 7.469, while the log-transformed standard deviation of the sample population (1.988) was obtained from Table B-1 (Appendix B).

Since the lead dataset had a lognormal distribution, VSP used the Wilcoxon Signed Rank Test to develop a performance curve. Using the interactive performance curve (Graph C-6, Appendix C), a lower boundary of the region of uncertainty was determined to be 7.21. This value was back-transformed from a log value that resulted in a FBAL of 1,350 mg/kg for lead. FPXRF lead concentrations below 1,350 mg/kg are expected to yield ICP results below the PRG of 1,754 mg/kg within the decision error rates (5 percent alpha, 10 percent beta) discussed above.

Arsenic

The region of uncertainty for arsenic was developed and set at 22 (an untransformed value), which resulted in a minimum required sample population of 606 samples. Reducing the region of uncertainty to 21 increased the required sample population from 606 to 664. The region of uncertainty for arsenic only

requires 606 samples to be analyzed. With 648 site-wide samples to be collected and analyzed, the width of the region for arsenic is conservative.

The statistical plots and the Shapiro-Wilk test (Graph C-4, Appendix C) indicated that the 120-second count homogenized FPXRF arsenic data set followed a normal distribution; therefore, untransformed values were used for VSP input. The untransformed value of the arsenic action level (200 mg/kg) was used, while the untransformed standard deviation of the sample population (184.716) was obtained from Table B-1 (Appendix B).

For arsenic, VSP used the One-Sample t-Test of the True Mean vs. the Action Level to develop a performance curve. The One-Sample t-Test of the True Mean vs. the Action level was used because the arsenic 120-second count homogenized data set follows a normal distribution. Using the interactive performance curve (Graph C-7, Appendix C), the FBAL was determined to be 177 mg/kg. XRF arsenic concentrations below 177 mg/kg are expected to yield ICP results below the PRG of 200 mg/kg within the decision error rates (5 percent alpha, 10 percent beta) discussed in previous text.

Antimony

The region of uncertainty for antimony was developed and set at 0.31 (a log transformed value), which resulted in a minimum required sample population of 626 samples. The region of uncertainty for antimony only requires 626 samples, so based on preliminary estimates (648 site-wide samples), the width of the region is conservative.

The statistical plots and the Shapiro-Wilk test (Graph C-5, Appendix C) indicated that the 120-second count homogenized XRF antimony data set followed a lognormal distribution; therefore, log-transformed values were used for VSP input. The log-transformed value of the antimony action level (310 mg/kg) was calculated to be 5.737, while the log-transformed standard deviation of the sample population (2.457) was taken from Table B-1 (Appendix B).

The Wilcoxon Signed Rank (One-Sample) Test was used because the antimony 120-second count homogenized data set follows a lognormal distribution. Using the interactive performance curve (Graph C-8, Appendix C), a lower boundary of the region of uncertainty was determined to be 5.42. When this value was back-transformed from a log value, it resulted in a FBAL of 225 mg/kg for antimony. XRF

15

antimony concentrations below 225 mg/kg are expected to yield ICP results below the PRG of 310 mg/kg within the decision error rates (5 percent alpha, 10 percent beta).

5.2   REFINING FIELD-BASED ACTION LEVELS

A common misconception is that the process is static and unchanging once the DMA is completed in the early portions of a project. However, results from the DMA can and should be continually refined as more data become available. For this site, the DMA was used to determine the best FPXRF operating procedures and preliminary FBAL for each of the three COCs. As additional FPXRF and ICP data becomes available, new summary statistics can be developed, likely reducing the standard deviation of the sample population. This reduction of the standard deviation and new estimates on actual samples expected to be collected and analyzed using FPXRF can be used to generate refined performance curves. Without changing any of the decision error rates but using only a higher sample population, lower standard deviations, new estimates of sample population sizes, and the region of uncertainty can be effectively reduced. A reduction in the region of uncertainty will actually slightly raise the FBALs without changing error tolerances or performance confidence. Higher FBALs may result in more efficient and accurate clean-up efforts reducing overall project costs.

REFERENCES

CH2M HILL. 2005a. "Final Basis of Design Report for Rockwool Industries, Inc., Belton, Texas." Prepared for EPA under Contract No. 68-W6-0036. January.

CH2M HILL. 2005b. "Addendum No. 1 to the Final Basis of Design Report." Rockwool Industries, Inc., Belton, Texas. Prepared for EPA under Contract No. 68-W6-0036. February.

Niton LLC Corporation. Available on-line at www.niton.com

Pacific Northwest National Laboratory. 2004. *Visual Sample Plan, Version 3.0*. Prepared for the U.S. Department of Energy and U.S. Environmental Protection Agency under Contract DE-AC05-76RL01830. Richland, WA. December. Available on line at http://dqo.pnl.gov/VSP/

Tetra Tech EM Inc. (Tetra Tech). 2005a. "Field Sampling Plan, Remedial Action, Rockwool Industries, Inc., Superfund Site, Belton, Texas." Prepared for EPA under Contract No. 68-W6-0037. April.

Tetra Tech. 2005b. "Quality Assurance Project Plan, Remedial Action, Rockwool Industries, Inc. Superfund Site, Belton, Texas." Prepared for EPA under Contract No. 68-W6-0037. April.

U.S. Environmental Protection Agency (EPA). 1996. *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*. SW-846. Third Edition. Update III. December.

EPA. 1998. *EPA SW-846 Method 6200–Field Portable X-Ray Fluorescence Spectrometry for Determination of Elemental Concentrations in Soil and Sediment.* Office of Solid Waste and Emergency Response. Washington, D.C. January.

APPENDIX A

XRF DATA SUMMARY TABLES

TABLE A-1

XRF DATA SUMMARY
EX SITU SAMPLE RESULTS
ROCKWOOL SUPERFUND SITE

Sample Location	Contaminant of Concern	Ex-Situ Homogenized		Ex-Situ Homogenized & Dried	
		60 NomSec Results	120 NomSec Results	60 NomSec Results	120 NomSec Results
DMA-NSP-C3-01	Lead (Pb)	165.0 ± 34.0	160.0 ± 26	223 ± 39	222 ± 29
	Arsenic (As)	53.4 ± 32.0	76.4 ± 25	962 ± 37	93 ± 27
	Antimony (Sb)	1820 ± 200	1720 ± 120	1890 ± 220	1920 ± 160
DMA-CP-J7-01	Lead (Pb)	<16	16.6 ± 8.0	24 ± 13	<14
	Arsenic (As)	<16	19.5 ± 8.1	<19	16 ± 9
	Antimony (Sb)	<42	<33	<50	<35
DMA-LRB-A-01	Lead (Pb)	664 ± 68	608 ± 45	733 ± 69	720 ± 49
	Arsenic (As)	199 ± 62	248 ± 43	188 ± 62	238 ± 46
	Antimony (Sb)	85 ± 56	145 ± 42	162 ± 68	124 ± 46
DMA-LRB-A-01-99	Lead (Pb)	666 ± 63	608 ± 45	1780 ± 110	1470 ± 74
	Arsenic (As)	219 ± 58	248 ± 43	234 ± 93	237 ± 62
	Antimony (Sb)	142 ± 66	137 ± 46	147 ± 69	148 ± 46
DMA-CSP-D2-01	Lead (Pb)	1010 ± 100	1220 ± 62	1370 ± 98	1270 ± 68
	Arsenic (As)	239 ± 100	319 ± 55	328 ± 85	436 ± 62
	Antimony (Sb)	<44	<34	202 ± 81	220 ± 57
DMA-SSP-E8-03	Lead (Pb)	<21	<16	58 ± 17	40.2 ± 11
	Arsenic (As)	<19	<15	<23	<16
	Antimony (Sb)	100 ± 53	112 ± 40	147 ± 54	142 ± 39
DMA-SSP-C9-04	Lead (Pb)	89.9 ± 21	104.0 ± 15	203 ± 32	213 ± 22
	Arsenic (As)	<29	<21	<44	49.8 ± 20
	Antimony (Sb)	801 ± 120	734 ± 79	1560 ± 170	1490 ± 120
DMA-SSP-C9-05	Lead (Pb)	1460 ± 82	1340 ± 57	1830 ± 94	1740 ± 64
	Arsenic (As)	329 ± 70	330 ± 49	538 ± 82	367 ± 54
	Antimony (Sb)	11500 ± 870	11100 ± 600	11400 ± 1000	10300 ± 560
DMA-SSP-D7-01	Lead (Pb)	51.8 ±15	36.2 ± 12	33.8 ± 16	50.9 ± 12
	Arsenic (As)	<25	<18	<24	18.5 ± 12
	Antimony (Sb)	83.5 ± 51	86.6 ± 37	73.3 ± 50	88.9 ± 35
DMA-BHD-I3-01	Lead (Pb)	2240 ± 97	2000 ± 65	2370 ± 100	2790 ± 77
	Arsenic (As)	532 ± 81	497 ± 55	598 ± 87	532 ± 62
	Antimony (Sb)	14000 ± 980	15000 ± 750	23500 ± 1800	21900 ± 1100
DMA-BHD-I3-02	Lead (Pb)	1840 ± 86	2030 ± 64	2790 ± 110	2830 ± 76
	Arsenic (As)	366 ± 72	445 ± 54	548 ± 85	497 ± 61
	Antimony (Sb)	6210 ± 470	6100 ± 330	7460 ± 610	8610 ± 490

TABLE A-2

XRF DATA SUMMARY
REPLICATE 01 RESULTS
ROCKWOOL SUPERFUND SITE

Sample Location	Contaminant of Concern	Ex-Situ Homogenized		Ex-Situ Homogenized & Dried	
		60 NomSec Results	120 NomSec Results	60 NomSec Results	120 NomSec Results
DMA-SSP-C9-05 (1)	Lead (Pb)	1260 ± 77	1300 +/- 55	2230 ± 100	2010 ± 68
	Arsenic (As)	272 ± 66	323 +/- 48	476 ± 84	474 ± 57
	Antimony (Sb)	9440 ± 730	9210 ± 510	13700 ± 1100	11900 ± 640
DMA-SSP-C9-05 (2)	Lead (Pb)	1430 ± 83	1320 +/- 55	2050 ± 95	2240 ± 72
	Arsenic (As)	266 ± 70	254 +/- 47	510 ± 80	438 ± 59
	Antimony (Sb)	9530 ± 780	9020 ± 500	12600 ± 960	12400 ± 680
DMA-SSP-C9-05 (3)	Lead (Pb)	1430 ± 80	1300 +/- 56	2110 ± 98	2120 ± 69
	Arsenic (As)	248 ± 67	309 +/- 48	484 ± 82	464 ± 58
	Antimony (Sb)	10400 ± 860	10200 ± 590	11300 ± 860	1200 ± 650
DMA-SSP-C9-05 (4)	Lead (Pb)	1370 ± 80	1350 +/- 52	2140 ± 100	2360 ± 79
	Arsenic (As)	357 ± 70	303 +/- 44	554 ± 98	543 ± 66
	Antimony (Sb)	10400 ± 870	9950 ± 560	12900 ± 980	9320 ± 500
DMA-SSP-C9-05 (5)	Lead (Pb)	1500 ± 86	1420 +/- 58	2130 ± 110	2120 ± 73
	Arsenic (As)	282 ± 72	327 +/- 50	567 ±	501 ± 62
	Antimony (Sb)	10600 ± 870	10800 ± 590	12600 ± 940	12100 ± 650
DMA-SSP-C9-05 (6)	Lead (Pb)	1530 ± 86	1400 ± 58	1520 ± 85	2360 ± 73
	Arsenic (As)	340 ± 74	294 ± 49	533 ± 31	532 ± 61
	Antimony (Sb)	10100 ± 700	10100 ± 550	13100 ± 950	12900 ± 720
DMA-SSP-C9-05 (7)	Lead (Pb)	1530 ± 86	1550 ± 62	2350 ± 100	2470 ± 73
	Arsenic (As)	295 ± 72	301 ± 52	651 ± 87	619 ± 61
	Antimony (Sb)	9820 ± 740	10500 ± 590	13300 ± 970	13200 ± 730
DMA-SSP-C9-05 (8)	Lead (Pb)	1600 ± 88	1510 ± 60	2600 ± 110	2520 ± 75
	Arsenic (As)	340 ± 75	362 ± 52	577 ± 89	606 ± 62
	Antimony (Sb)	10700 ± 830	10600 ± 590	12800 ± 950	11700 ± 610
DMA-SSP-C9-05 (9)	Lead (Pb)	1520 ± 86	1450 ± 58	1930 ± 93	2050 ± 68
	Arsenic (As)	270 ± 72	290 ± 49	520 ± 80	452 ± 57
	Antimony (Sb)	10700 ± 880	10300 ± 590	12200 ± 890	12600 ± 670
DMA-SSP-C9-05 (10)	Lead (Pb)	1410 ± 84	1370 ± 58	2060 ± 95	2320 ± 72
	Arsenic (As)	310 ± 72	363 ± 50	504 ± 80	552 ± 60
	Antimony (Sb)	9820 ± 830	10000 ± 580	12600 ± 950	13300 ± 710
Standard Deviation (Pb) =		98.32	476.34	278.75	415.49
Mean (Pb) =		1457.76	1676.72	2111.36	1976.88
Relative Standard Deviation (Pb) =		6.74	28.41	13.20	21.02
Standard Deviation (As) =		37.21	109.88	88.32	120.91
Mean (As) =		298.12	386.64	515.62	444.04
Relative Standard Deviation (As) =		12.48	28.42	17.13	27.23
Standard Deviation (Sb) =		1184.61	1466.65	1368.93	1328.82
Mean (Sb) =		10755.20	10875.52	12100.48	11349.12
Relative Standard Deviation (Sb) =		11.01	13.49	11.31	11.71

XRF DATA SUMMARY
REPLICATE 02 RESULTS
ROCKWOOL SUPERFUND SITE

Sample Location	Contaminant of Concern	Ex-Situ Homogenized		Ex-Situ Homogenized & Dried	
		60 NomSec Results	120 NomSec Results	60 NomSec Results	120 NomSec Results
DMA-BHD-I3-01 (1)	Lead (Pb)	2050 ± 88	2360 ± 72	1900 ± 84	2040 ± 57
	Arsenic (As)	489 ± 74	522 ± 60	414 ± 71	389 ± 47
	Antimony (Sb)	21800 ± 1600	22100 ± 1100	18400 ± 1400	18600 ± 980
DMA-BHD-I3-01 (2)	Lead (Pb)	1930 ± 89	2350 ± 72	2060 ± 89	1950 ± 63
	Arsenic (As)	436 ± 75	507 ± 59	358 ± 74	398 ± 53
	Antimony (Sb)	21200 ± 1500	22100 ± 990	17200 ± 1200	18300 ± 940
DMA-BHD-I3-01 (3)	Lead (Pb)	2090 ± 85	2320 ± 72	2050 ± 91	1880 ± 63
	Arsenic (As)	448 ± 71	494 ± 59	274 ± 74	395 ± 53
	Antimony (Sb)	22900 ± 1600	23100 ± 1200	18100 ± 1300	17900 ± 940
DMA-BHD-I3-01 (4)	Lead (Pb)	2190 ± 97	2370 ± 72	1850 ± 83	2000 ± 64
	Arsenic (As)	368 ± 80	439 ± 59	323 ± 69	379 ± 53
	Antimony (Sb)	21600 ± 1500	22200 ± 1100	18200 ± 1400	17100 ± 860
DMA-BHD-I3-01 (5)	Lead (Pb)	2200 ± 97	2380 ± 71	1900 ± 87	1950 ± 62
	Arsenic (As)	428 ± 80	525 ± 59	310 ± 72	339 ± 52
	Antimony (Sb)	21600 ± 1500	21800 ± 1100	16000 ± 1100	17600 ± 930
DMA-BHD-I3-01 (6)	Lead (Pb)	2280 ± 99	2280 ± 71	1900 ± 83	1950 ± 62
	Arsenic (As)	544 ± 83	475 ± 59	364 ± 70	329 ± 51
	Antimony (Sb)	21300 ± 1500	22000 ± 1100	17300 ± 1300	16800 ± 860
DMA-BHD-I3-01 (7)	Lead (Pb)	2490 ± 95	1860 ± 64	1660 ± 78	1630 ± 55
	Arsenic (As)	457 ± 77	387 ± 53	325 ± 66	339 ± 47
	Antimony (Sb)	21100 ± 1500	22200 ± 1100	16300 ± 1200	17000 ± 1870
DMA-BHD-I3-01 (8)	Lead (Pb)	2530 ± 110	1930 ± 55	1630 ± 71	1640 ± 56
	Arsenic (As)	402 ± 85	382 ± 46	242 ± 59	290 ± 47
	Antimony (Sb)	20100 ± 1400	21800 ± 1100	15800 ± 1100	15800 ± 820
DMA-BHD-I3-01 (9)	Lead (Pb)	2420 ± 100	2040 ± 65	2060 ± 98	2029 ± 69.4
	Arsenic (As)	387 ± 83	398 ± 54	483 ± 83	422 ± 58
	Antimony (Sb)	20200 ± 1300	21500 ± 1100	15800 ± 1200	15900 ± 870
DMA-BHD-I3-01 (10)	Lead (Pb)	2400 ± 100	1990 ± 64	1860 ± 91	1400 ± 54
	Arsenic (As)	431 ± 82	422 ± 54	373 ± 76	309 ± 47
	Antimony (Sb)	21200 ± 1500	21700 ± 1100	19700 ± 1500	17400 ± 920
Standard Deviation (Pb) =		200.51	288.13	151.48	233.65
Mean (Pb) =		2257.60	2102.48	1886.32	1931.52
Relative Standard Deviation (Pb) =		8.88	13.70	8.03	12.10
Standard Deviation (As) =		50.93	75.20	69.01	57.82
Mean (As) =		439.08	432.16	346.64	383.92
Relative Standard Deviation (As) =		11.60	17.40	19.91	15.06
Standard Deviation (Sb) =		2403.33	1899.00	2337.00	2478.87
Mean (Sb) =		19516.24	21004.80	19055.36	18274.56
Relative Standard Deviation (Sb) =		12.31	9.04	12.26	13.56

APPENDIX B

DMA STATISICAL DATA TABLES

Rockwool Homogenized Sample XRF Analyses
60 and 120 Nominal Count Times

Analyte	Count Time	Valid N	Mean	Confidence -95.000%	Confidence +95.000%	Geometric Mean	Harmonic Mean	Median	Mode	Frequency of Mode	Sum	Minimum	Maximum	Lower Quartile	Upper Quartile	Percentile 10.00000	Percentile 90.00000	Range	Quartile Range	Variance	Standard Deviation	Standard Error	Skewness	Std. Error Skewness	Kurtosis	Std.Erroe Kurtosis
Lead	60 Sec	11	745.9273	210.2871	1281.567	242.8762	41.92079	664.0000	Multiple	1	8205.200	8.000000	2240.000	51.80000	1460.000	10.50000	1840.000	2232.000	1408.200	635703.4	797.3101	240.3980	0.823201	0.660687	-0.627652	1.279416
Lead	120 Sec	11	739.1636	211.3635	1266.964	245.6178	46.89258	608.0000	608	2	8130.800	8.000000	2030.000	36.20000	1340.000	16.60000	2000.000	2022.000	1303.800	617230.2	785.6400	236.8794	0.734152	0.660687	-1.00704	1.279416
Lead Logtransformed	60 Sec	11	5.4926	4.1264	6.859	5.0523	4.52711	6.4983	Multiple	1	60.418	2.079442	7.714	3.94739	7.286	2.35138	7.518	5.635	3.339	4.1	2.0336	0.6131	-0.661336	0.660687	-0.977772	1.279416
Lead Logtransformed	120 Sec	11	5.5038	4.1682	6.839	5.0957	4.61741	6.4102	6.410175	2	60.542	2.079442	7.616	3.58906	7.200	2.80940	7.601	5.536	3.611	4.0	1.9881	0.5994	-0.595945	0.660687	-1.11876	1.279416
Arsenic	60 Sec	11	180.1727	60.71305	299.6324	76.03130	26.22906	199.0000	Multiple	1	1981.900	8.000000	532.0000	12.50000	329.0000	9.500000	366.0000	524.0000	316.5000	31619.20	177.8179	53.61411	0.699062	0.660687	-0.39853	1.279416
Arsenic	120 Sec	11	200.9000	76.80632	324.9937	84.21961	26.03289	248.0000	248	2	2209.900	7.500000	497.0000	10.50000	330.0000	9.000000	445.0000	489.5000	319.5000	34119.88	184.7157	55.69387	0.298864	0.660687	-1.47188	1.279416
Arsenic Logtransformed	60 Sec	11	4.3311	3.22148	5.4408	4.00216	3.66093	5.2933	Multiple	1	47.643	2.079442	6.2766	2.52573	5.7961	2.251292	5.9026	4.1972	3.2703	2.73	1.6518	0.49803	-0.350584	0.660687	-1.91413	1.279416
Arsenic Logtransformed	120 Sec	11	4.4334	3.28488	5.5820	4.07598	3.69414	5.5134	5.513429	2	48.768	2.014903	6.2086	2.35138	5.7991	2.197225	6.0981	4.1937	3.4477	2.92	1.7096	0.51547	-0.485558	0.660687	-1.81578	1.279416
Antimony	60 Sec	11	3162.227	-268.718	6593.172	417.8878	80.89787	142.0000	Multiple	1	34784.50	21.00000	14000.00	83.50000	6210.000	22.00000	11500.00	13979.00	6126.500	26081689	5107.024	1539.826	1.549605	0.660687	1.03344	1.279416
Antimony	120 Sec	11	3197.100	-335.758	6729.958	418.6819	70.34786	145.0000	Multiple	1	35168.10	16.50000	15000.00	86.60000	6100.000	17.00000	11100.00	14983.50	6013.400	27654162	5258.722	1585.564	1.649997	0.660687	1.58285	1.279416
Antimony Logtransformed	60 Sec	11	6.035	4.401	7.669	5.5859	5.16197	4.9558	Multiple	1	66.39	3.04452	9.55	4.42485	8.734	3.09104	9.35	6.50	4.309	6	2.432	0.733	0.324408	0.660687	-1.52221	1.279416
Antimony Logtransformed	120 Sec	11	6.037	4.387	7.688	5.5605	5.08835	4.9767	Multiple	1	66.41	2.80336	9.62	4.46130	8.716	2.83321	9.31	6.81	4.255	6	2.457	0.741	0.228910	0.660687	-1.33942	1.279416

Table B-2

Rockwool Dried and Homogenized Sample XRF Analyses

60 and 120 Second Nominal Count Times

Analyte	Count Time	Valid N	Mean	Confidence -95.000%	Confidence +95.000%	Geometric Mean	Harmonic Mean	Median	Mode	Frequency of Mode	Sum	Minimum	Maximum	Lower Quartile	Upper Quartile	Percentile 10.00000	Percentile 90.00000	Range	Quartile Range	Variance	Std.Dev.	Standard Error	Skewness	Std. Error Skewness	Kurtosis	Std.Error Kurtosis
Lead	60 Sec	11	1037.709	346.5854	1728.833	403.4262	107.9627	733.0000	Multiple	1	11414.80	24.00000	2790.000	58.00000	1830.000	33.80000	2370.000	2766.000	1772.000	1058327	1028.750	310.1798	0.510087	0.660687	-1.34868	1.279416
Lead	120 Sec	11	1032.100	311.8475	1752.353	358.3340	54.80229	720.0000	Multiple	1	11353.10	7.000000	2830.000	50.90000	1740.000	40.20000	2790.000	2823.000	1689.100	1149417	1072.109	323.2530	0.752468	0.660687	-0.811471	1.279416
Lead Logtransformed	60 Sec	11	6.000	4.7999	7.200	5.7226	5.4197	6.5971	Multiple	1	66.00	3.17805	7.934	4.06044	7.512	3.52046	7.771	4.756	3.452	3	1.786	0.5386	-0.514418	0.660687	-1.43088	1.279416
Lead Logtransformed	120 Sec	11	5.881	4.5439	7.219	5.4732	4.94247	6.5793	Multiple	1	64.70	1.945910	7.948	3.92986	7.462	3.69387	7.934	6.002	3.532	4	1.991	0.6003	-0.813179	0.660687	-0.358786	1.279416
Arsenic	60 Sec	11	313.7273	102.4908	524.9637	119.4416	32.34873	234.0000	Multiple	1	3451.000	9.500000	962.0000	12.00000	548.0000	11.50000	598.0000	952.5000	536.0000	98865.77	314.4293	94.80399	0.821731	0.660687	-0.01363	1.279416
Arsenic	120 Sec	11	226.5727	89.75849	363.3870	109.1404	37.96792	237.0000	Multiple	1	2492.300	8.000000	532.0000	18.50000	436.0000	16.00000	497.0000	524.0000	417.5000	41473.51	203.6505	61.40292	0.340519	0.660687	-1.63199	1.279416
Arsenic Logtransformed	60 Sec	11	4.7828	3.5580	6.0077	4.4068	4.00572	5.4553	Multiple	1	52.611	2.251292	6.8690	2.48491	6.3063	2.44235	6.3936	4.6177	3.8214	3.32	1.8232	0.54971	-0.491863	0.660687	-1.76687	1.279416
Arsenic Logtransformed	120 Sec	11	4.6926	3.65814	5.7271	4.4165	4.10342	5.4681	Multiple	1	51.619	2.079442	6.2766	2.91777	6.0776	2.77259	6.2086	4.1972	3.1599	2.37	1.5399	0.46429	-0.607967	0.660687	-1.27523	1.279416
Antimony	60 Sec	11	4233.300	-734.702	9201.302	641.9943	137.8346	202.0000	147.0000	2	46566.30	25.00000	23500.00	147.0000	7460.000	73.30000	11400.00	23475.00	7313.000	54685445	7394.961	2229.665	2.147563	0.660687	4.55897	1.279416
Antimony	120 Sec	11	4087.309	-577.222	8751.840	617.2544	114.2842	220.0000	Multiple	1	44960.40	17.50000	21900.00	124.0000	8610.000	88.90000	10300.00	21882.50	8486.000	48208560	6943.238	2093.465	2.038400	0.660687	4.04787	1.279416
Antimony Logtransformed	60 Sec	11	6.465	4.932	7.997	6.0965	5.7401	5.3083	4.990433	2	71.11	3.21888	10.06	4.9904	8.917	4.29456	9.34	6.85	3.927	5	2.281	0.688	0.331714	0.660687	-1.30448	1.279416
Antimony Logtransformed	120 Sec	11	6.425	4.865	7.986	6.0285	5.6273	5.3936	Multiple	1	70.68	2.86220	9.99	4.8203	9.061	4.48751	9.24	7.13	4.240	5	2.323	0.700	0.237707	0.660687	-1.22376	1.279416

Table B-3

Rockwool Homogenized vs. Dried/Homogenized Sample Analyses

60 and 120 Second Nominal Count Times Combined for Evaluation of Variance Associated with Preparation Method

Analyte	Sample Prep	Valid N	Mean	Confidence -95.000%	Confidence +95.000%	Geometric Mean	Harmonic Mean	Median	Mode	Frequency of Mode	Sum	Minimum	Maximum	Lower Quartile	Upper Quartile	Percentile 10.00000	Percentile 90.00000	Range	Quartile Range	Variance	Std.Dev.	Standard Error	Skewness	Std. Error Skewness	Kurtosis	Std.Erroe Kurtosis
Lead	Homogenized	22	742.5455	400.0695	1085.021	244.2432	44.26752	608.0000	Multiple	2	16336.00	8.000000	2240.000	51.80000	1340.000	10.50000	2000.000	2232.000	1288.200	596647.0	772.4293	164.6825	0.719274	0.490962	-0.92120	0.952780
Lead	Homogenized/Dried	22	1034.905	580.2958	1489.513	380.2122	72.70118	726.5000	2790.000	2	22767.90	7.000000	2830.000	58.00000	1780.000	33.80000	2790.000	2823.000	1722.000	1051315	1025.337	218.6025	0.589089	0.490962	-1.10867	0.952780
Lead Logtransformed	Homogenized	22	5.4982	4.6280	6.368	5.0740	4.57181	6.4102	Multiple	2	120.96	2.079442	7.714	3.94739	7.200	2.35138	7.601	5.635	3.253	3.9	1.9625	0.4184	-0.581090	0.490962	-1.09902	0.952780
Lead Logtransformed	Homogenized/Dried	22	5.941	5.1219	6.760	5.5965	5.17009	6.5882	7.933797	2	130.70	1.945910	7.948	4.06044	7.484	3.52046	7.934	6.002	3.424	3	1.847	0.3938	-0.647565	0.490962	-0.82949	0.952780
Arsenic	Homogenized	22	190.5364	111.9490	269.1237	80.02079	26.13061	209.0000	248.0000	2	4191.800	7.500000	532.0000	12.50000	329.0000	9.000000	445.0000	524.5000	316.5000	31416.84	177.2480	37.78939	0.454776	0.490962	-1.07953	0.952780
Arsenic	Homogenized/Dried	22	270.1500	153.8389	386.4611	114.1749	34.93380	235.5000	Multiple	1	5943.300	8.000000	962.0000	18.50000	497.0000	11.50000	548.0000	954.0000	478.5000	68817.63	262.3312	55.92919	0.892395	0.490962	0.44942	0.952780
Arsenic Logtransformed	Homogenized	22	4.3823	3.6546	5.1100	4.03890	3.67746	5.3412	5.513429	2	96.410	2.014903	6.2766	2.52573	5.7961	2.197225	6.0981	4.2617	3.2703	2.69	1.6413	0.34992	-0.384811	0.490962	-1.72977	0.952780
Arsenic Logtransformed	Homogenized/Dried	22	4.7377	4.0073	5.4682	4.4116	4.05398	5.4617	Multiple	1	104.230	2.079442	6.8690	2.91777	6.2086	2.44235	6.3063	4.7896	3.2908	2.71	1.6475	0.35124	-0.483370	0.490962	-1.47705	0.952780
Antimony	Homogenized	22	3179.664	936.8330	5422.494	418.2847	75.25491	143.5000	Multiple	1	69952.60	16.50000	15000.00	85.00000	6100.000	21.00000	11500.00	14983.50	6015.000	25588819	5058.539	1078.484	1.478188	0.490962	0.71137	0.952780
Antimony	Homogenized/Dried	22	4160.305	1056.592	7264.017	629.5028	124.9595	211.0000	147.0000	2	91526.70	17.50000	23500.00	142.0000	7460.000	73.30000	11400.00	23482.50	7318.000	49002727	7000.195	1492.447	1.939657	0.490962	3.03924	0.952780
Antimony Logtransformed	Homogenized	22	6.036	4.9785	7.094	5.5732	5.12489	4.9663	Multiple	1	132.80	2.80336	9.62	4.44265	8.716	3.04452	9.35	6.81	4.273	6	2.385	0.509	0.254496	0.490962	-1.39358	0.952780
Antimony Logtransformed	Homogenized/Dried	22	6.445	5.449	7.441	6.0624	5.6831	5.3509	4.990433	2	141.79	2.86220	10.06	4.9558	8.917	4.29456	9.34	7.20	3.961	5	2.247	0.479	0.260860	0.490962	-1.26436	0.952780

Table B-4

Rockwool Homogenized and Dried Homogenized Combined XRF Analyses

60 and 120 Second Nominal Count Times Separated for Evaluation of Variance Associated with Count Time
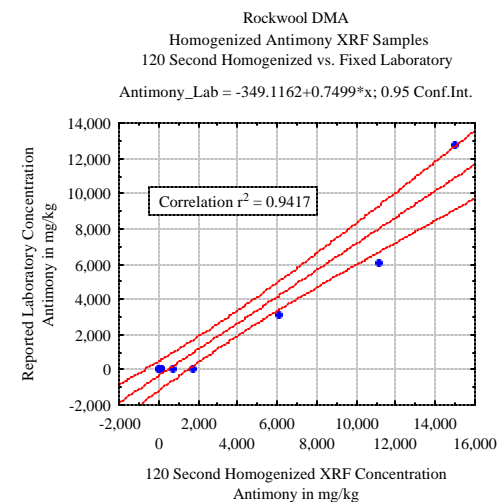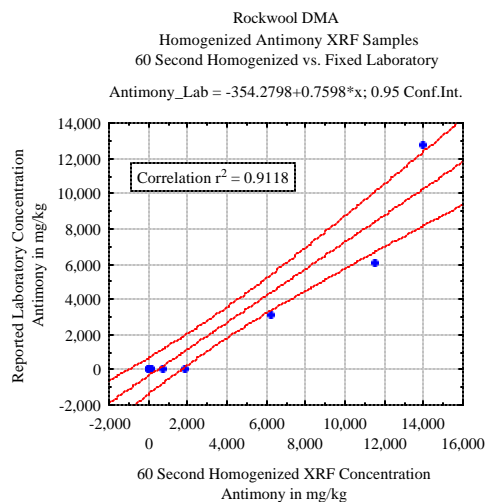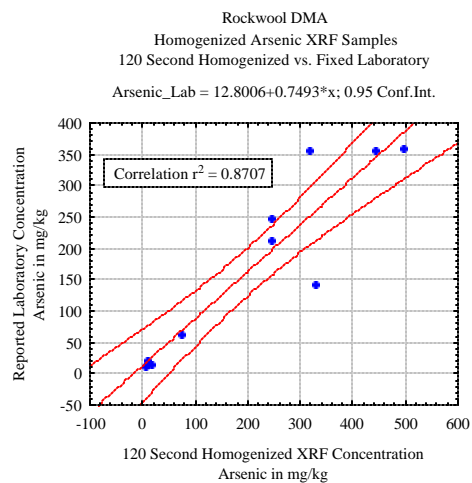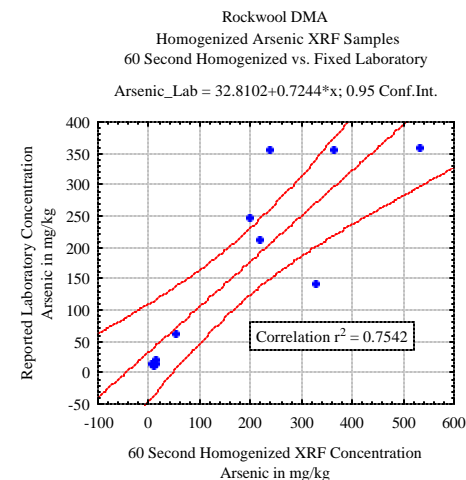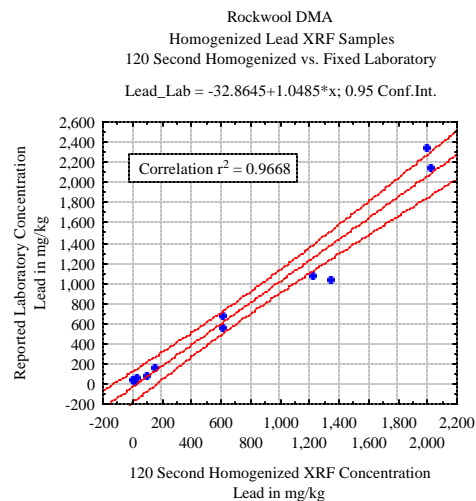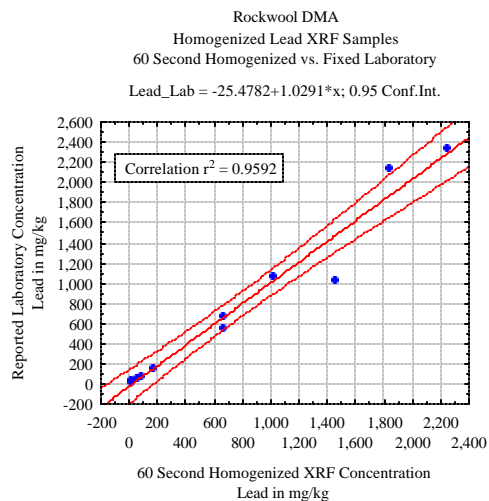
Analyte	Count Time	Valid N	Mean	Confidence -95.000%	Confidence +95.000%	Geometric Mean	Harmonic Mean	Median	Mode	Frequency of Mode	Sum	Minimum	Maximum	Lower Quartile	Upper Quartile	Percentile 10.00000	Percentile 90.00000	Range	Quartile Range	Variance	Std.Dev.	Standard Error	Skewness	Std. Error Skewness	Kurtosis	Std.Erroe Kurtosis
Lead	60 Sec	22	891.8182	488.1328	1295.504	313.0218	60.39199	665.0000	Multiple	1	19620.00	8.000000	2790.000	58.00000	1780.000	24.00000	2240.000	2782.000	1722.000	828978.7	910.4827	194.1156	0.680628	0.490962	-0.911862	0.952780
Lead	120 Sec	22	885.6318	473.5706	1297.693	296.6702	50.53983	608.0000	608.0000	2	19483.90	7.000000	2830.000	50.90000	1470.000	16.60000	2030.000	2823.000	1419.100	863735.3	929.3736	198.1431	0.837719	0.490962	-0.441620	0.952780
Lead Logtransformed	60 Sec	22	5.7463	4.9101	6.582	5.3770	4.93335	6.4998	Multiple	1	126.42	2.079442	7.934	4.06044	7.484	3.17805	7.714	5.854	3.424	3.6	1.8858	0.4021	-0.598943	0.490962	-0.976514	0.952780
Lead Logtransformed	120 Sec	22	5.6926	4.8275	6.558	5.2811	4.77441	6.4102	6.410175	2	125.24	1.945910	7.948	3.92986	7.293	2.80940	7.616	6.002	3.363	3.8	1.9512	0.4160	-0.639971	0.490962	-0.890260	0.952780
Arsenic	60 Sec	22	246.9500	132.3504	361.5496	95.29586	28.96923	209.0000	9.500000	2	5432.900	8.000000	962.0000	12.50000	366.0000	9.500000	548.0000	954.0000	353.5000	66807.25	258.4710	55.10620	1.140203	0.490962	1.12934	0.952780
Arsenic	120 Sec	22	213.7364	129.4141	298.0587	95.87367	30.88756	237.5000	Multiple	2	4702.200	7.500000	532.0000	18.50000	367.0000	9.000000	497.0000	524.5000	348.5000	36169.47	190.1827	40.54709	0.318104	0.490962	-1.44310	0.952780
Arsenic Logtransformed	60 Sec	22	4.5570	3.7973	5.3166	4.19960	3.82557	5.3412	2.251292	2	100.254	2.079442	6.8690	2.52573	5.9026	2.251292	6.3063	4.7896	3.3769	2.94	1.7133	0.36528	-0.346597	0.490962	-1.68383	0.952780
Arsenic Logtransformed	120 Sec	22	4.5630	3.8566	5.2695	4.24284	3.88804	5.4702	Multiple	2	100.387	2.014903	6.2766	2.91777	5.9054	2.197225	6.2086	4.2617	2.9876	2.54	1.5933	0.33969	-0.520088	0.490962	-1.47000	0.952780
Antimony	60 Sec	22	3697.764	937.3839	6458.143	517.9591	101.9558	182.0000	147.0000	2	81350.80	21.00000	23500.00	85.00000	6210.000	25.00000	11500.00	23479.00	6125.000	38760996	6225.833	1327.352	2.008674	0.490962	3.88820	0.952780
Antimony	120 Sec	22	3642.205	969.6908	6314.718	508.3633	87.08832	184.0000	Multiple	1	80128.50	16.50000	21900.00	112.0000	6100.000	17.50000	11100.00	21883.50	5988.000	36332658	6027.658	1285.101	1.870439	0.490962	3.02533	0.952780
Antimony Logtransformed	60 Sec	22	6.250	5.2251	7.275	5.8356	5.4357	5.1979	4.990433	2	137.50	3.04452	10.06	4.44265	8.734	3.21888	9.35	7.02	4.291	5	2.311	0.493	0.279160	0.490962	-1.36456	0.952780
Antimony Logtransformed	120 Sec	22	6.231	5.1930	7.269	5.7898	5.34426	5.1954	Multiple	1	137.09	2.80336	9.99	4.7185	8.716	2.86220	9.31	7.19	3.998	5	2.342	0.499	0.197565	0.490962	-1.26196	0.952780

APPENDIX C
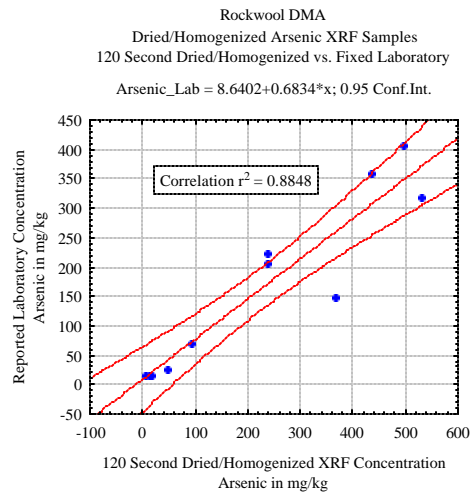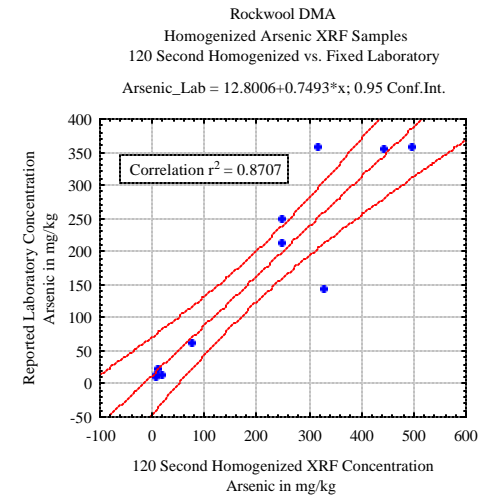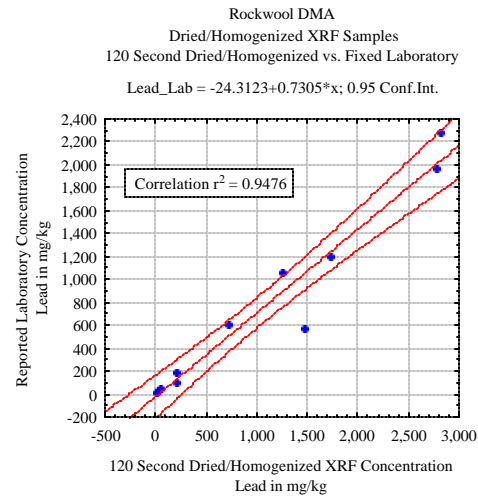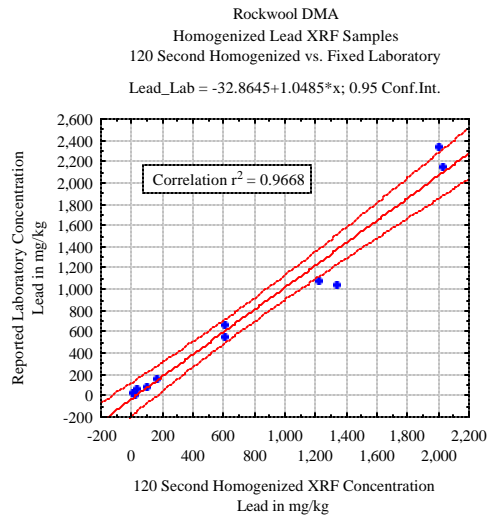
DMA STATISICAL DATA GRAPHS

Rockwool DMA
Homogenized XRF Lead, Arsenic, and Antimony
60 and 120 Second XRF Analyses vs. Fixed Laboratory

Rockwool DMA
Homogenized Lead XRF Samples
60 Second Homogenized vs. Fixed Laboratory

Lead_Lab = -25.4782+1.0291*x; 0.95 Conf.Int.

Rockwool DMA
Homogenized Lead XRF Samples
120 Second Homogenized vs. Fixed Laboratory

Lead_Lab = -32.8645+1.0485*x; 0.95 Conf.Int.

Rockwool DMA
Homogenized Arsenic XRF Samples
60 Second Homogenized vs. Fixed Laboratory

Arsenic_Lab = 32.8102+0.7244*x; 0.95 Conf.Int.

Rockwool DMA
Homogenized Arsenic XRF Samples
120 Second Homogenized vs. Fixed Laboratory

Arsenic_Lab = 12.8006+0.7493*x; 0.95 Conf.Int.

Rockwool DMA
Homogenized Antimony XRF Samples
60 Second Homogenized vs. Fixed Laboratory

Antimony_Lab = -354.2798+0.7598*x; 0.95 Conf.Int.

Rockwool DMA
Homogenized Antimony XRF Samples
120 Second Homogenized vs. Fixed Laboratory

Antimony_Lab = -349.1162+0.7499*x; 0.95 Conf.Int.

Rockwool DMA
120 Second XRF Lead, Arsenic, and Antimony
Homogenized and Dried/Homogenized vs. Fixed Laboratory

Rockwool DMA
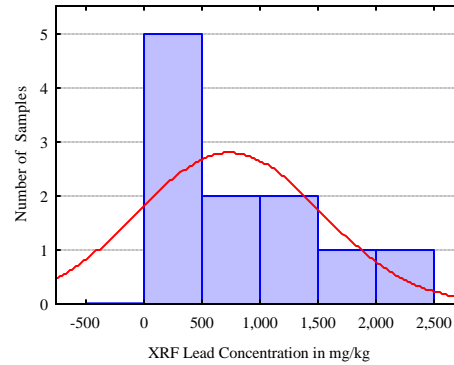Homogenized Lead XRF Samples
120 Second Homogenized vs. Fixed Laboratory

Lead_Lab = -32.8645+1.0485*x; 0.95 Conf.Int.

Correlation r^2 = 0.9668

Reported Laboratory Concentration Lead in mg/kg

120 Second Homogenized XRF Concentration
Lead in mg/kg

Rockwool DMA
Dried/Homogenized XRF Samples
120 Second Dried/Homogenized vs. Fixed Laboratory

Lead_Lab = -24.3123+0.7305*x; 0.95 Conf.Int.

Correlation r^2 = 0.9476

Reported Laboratory Concentration Lead in mg/kg

120 Second Dried/Homogenized XRF Concentration
Lead in mg/kg

Rockwool DMA
Homogenized Arsenic XRF Samples
120 Second Homogenized vs. Fixed Laboratory

Arsenic_Lab = 12.8006+0.7493*x; 0.95 Conf.Int.

Correlation r^2 = 0.8707

Reported Laboratory Concentration Arsenic in mg/kg

120 Second Homogenized XRF Concentration
Arsenic in mg/kg

Rockwool DMA
Dried/Homogenized Arsenic XRF Samples
120 Second Dried/Homogenized vs. Fixed Laboratory

Arsenic_Lab = 8.6402+0.6834*x; 0.95 Conf.Int.

Correlation r^2 = 0.8848

Reported Laboratory Concentration Arsenic in mg/kg

120 Second Dried/Homogenized XRF Concentration
Arsenic in mg/kg

Rockwool DMA
Homogenized Antimony XRF Samples
120 Second Homogenized vs. Fixed Laboratory

Antimony_Lab = -349.1162+0.7499*x; 0.95 Conf.Int.

Correlation r^2 = 0.9417

Reported Laboratory Concentration Antimony in mg/kg

120 Second Homogenized XRF Concentration
Antimony in mg/kg

Rockwool DMA
Dried/Homogenized Antimony XRF Samples
120 Second Dried/Homogenized vs. Fixed Laboratory

Antimony_Lab = -84.1588+0.5156*x; 0.95 Conf.Int.

Correlation r^2 = 0.9783

Reported Laboratory Concentration Antimony in mg/kg

120 Second Dried/Homogenized XRF Concentration
Antimony in mg/kg

Rockwool DMA
Lead Concentrations in mg/kg
120 Second Homogenized XRF Samples

Rockwool DMA
Box and Whisker Plots for Lead
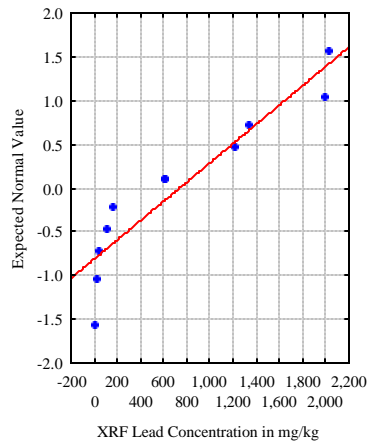120 Second Homogenized XRF Samples

- Mean
- Mean±SD
- Mean±1.96*SD

Untransformed Data
Logtransformed Data

Rockwool DMA
Histogram Untransformed Data
120 Second Homogenized XRF Samples
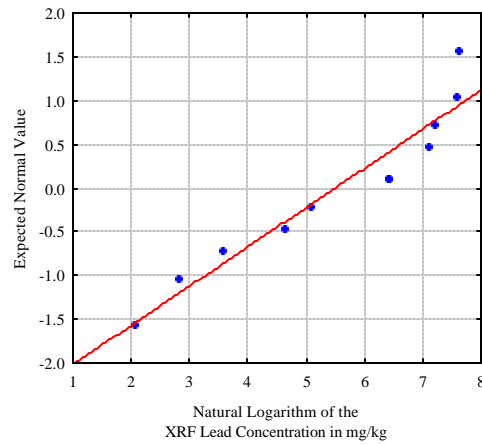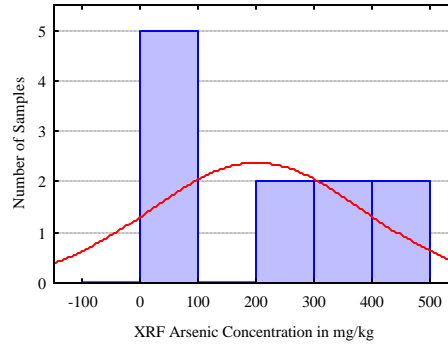K-S d=.22404, p> .20; Lilliefors p<.15
Shapiro-Wilk W=.83904, p=.03068

XRF Lead Concentration in mg/kg

Rockwool DMA
Histogram Logtransformed Data
120 Second Homogenized XRF Samples
K-S d=.22123, p> .20; Lilliefors p<.15
Shapiro-Wilk W=.89604, p=.16530

Natural Logarithm of the
XRF Lead Concentration in mg/kg

Rockwool DMA
Normal Probability Plot
120 Second Homogenized XRF Samples

XRF Lead Concentration in mg/kg

Rockwool DMA
Lognormal Probability Plot
120 Second Homogenized XRF Samples

Natural Logarithm of the
XRF Lead Concentration in mg/kg

Rockwool DMA
Arsenic Concentrations in mg/kg
120 Second Homogenized XRF Samples

Rockwool DMA
Box and Whisker Plots for Arsenic
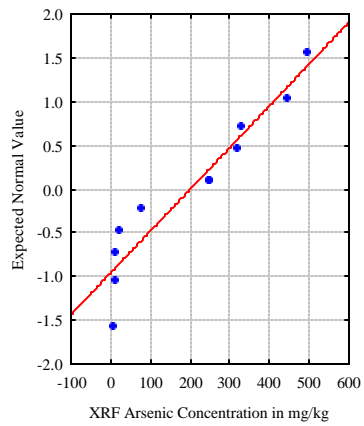120 Second Homogenized XRF Samples
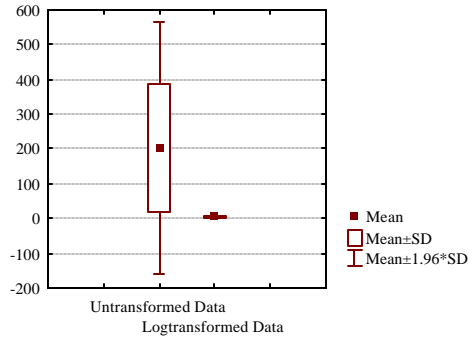
Rockwool DMA
Histogram Untransformed Data
120 Second Homogenized XRF Samples
Arsenic in mg/kg

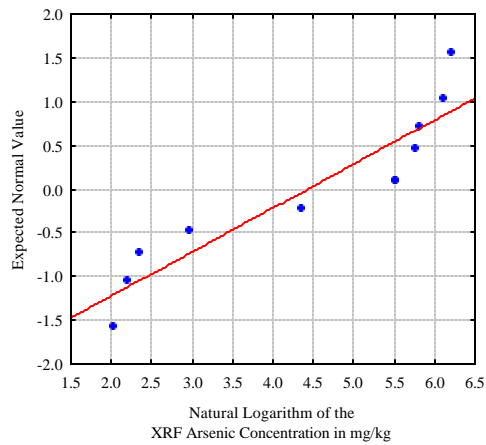K-S d=.20439, p> .20; Lilliefors p> .20

Shapiro-Wilk W=.87297, p=.08464

Rockwool DMA
Histogram Logtransformed Data
120 Second Homogenized XRF Samples
Arsenic in mg/kg

K-S d=.28167, p> .20; Lilliefors p<.05

Shapiro-Wilk W=.82264, p=.01869
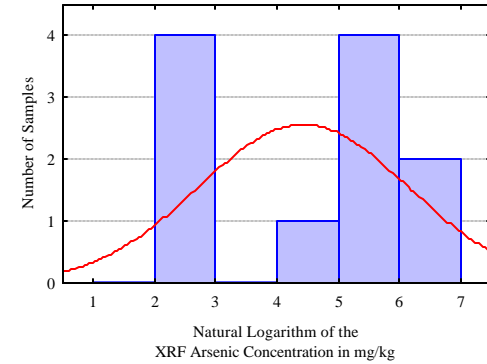
- Mean
- Mean±SD
- Mean±1.96*SD

Untransformed Data
Logtransformed Data

Number of Samples

XRF Arsenic Concentration in mg/kg

Number of Samples

Natural Logarithm of the
XRF Arsenic Concentration in mg/kg

Rockwool DMA
Normal Probability Plot
120 Second Homogenized XRF Samples

Rockwool DMA
Lognormal Probability Plot
120 Second Homogenized XRF Samples

Expected Normal Value

XRF Arsenic Concentration in mg/kg

Expected Normal Value

Natural Logarithm of the
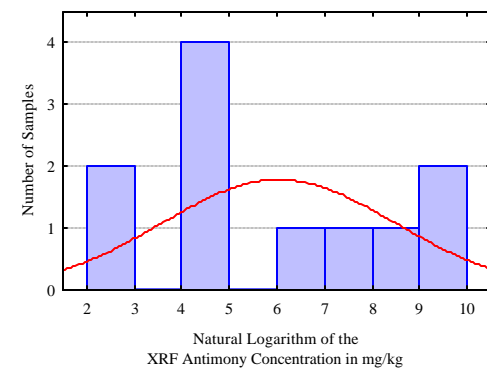XRF Arsenic Concentration in mg/kg

Rockwool DMA
Antimony Concentrations in mg/kg
120 Second Homogenized XRF Samples

Rockwool DMA
Box and Whisker Plots for Antimony
120 Second Homogenized XRF Samples
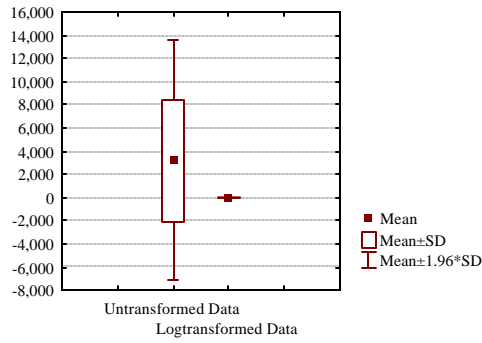
- ■ Mean
- ☐ Mean±SD
- I Mean±1.96*SD

Untransformed Data
Logtransformed Data

Rockwool DMA
Histogram Untransformed Data
120 Second Homogenized XRF Samples
Antimony in mg/kg
K-S d=.33787, p<.15 ; Lilliefors p<.01
Shapiro-Wilk W=.67834, p=.00025
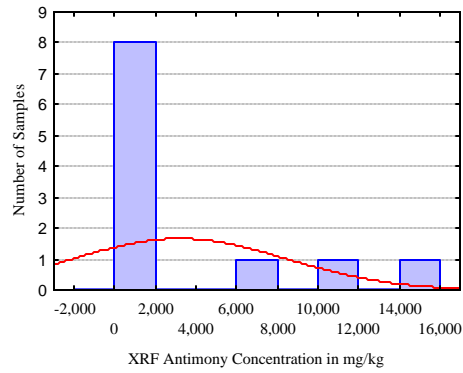
Number of Samples

XRF Antimony Concentration in mg/kg

Rockwool DMA
Histogram Logtransformed Data
120 Second Homogenized XRF Samples
Antimony in mg/kg
K-S d=.21245, p> .20; Lilliefors p<.20
Shapiro-Wilk W=.91563, p=.28394

Number of Samples

Natural Logarithm of the
XRF Antimony Concentration in mg/kg
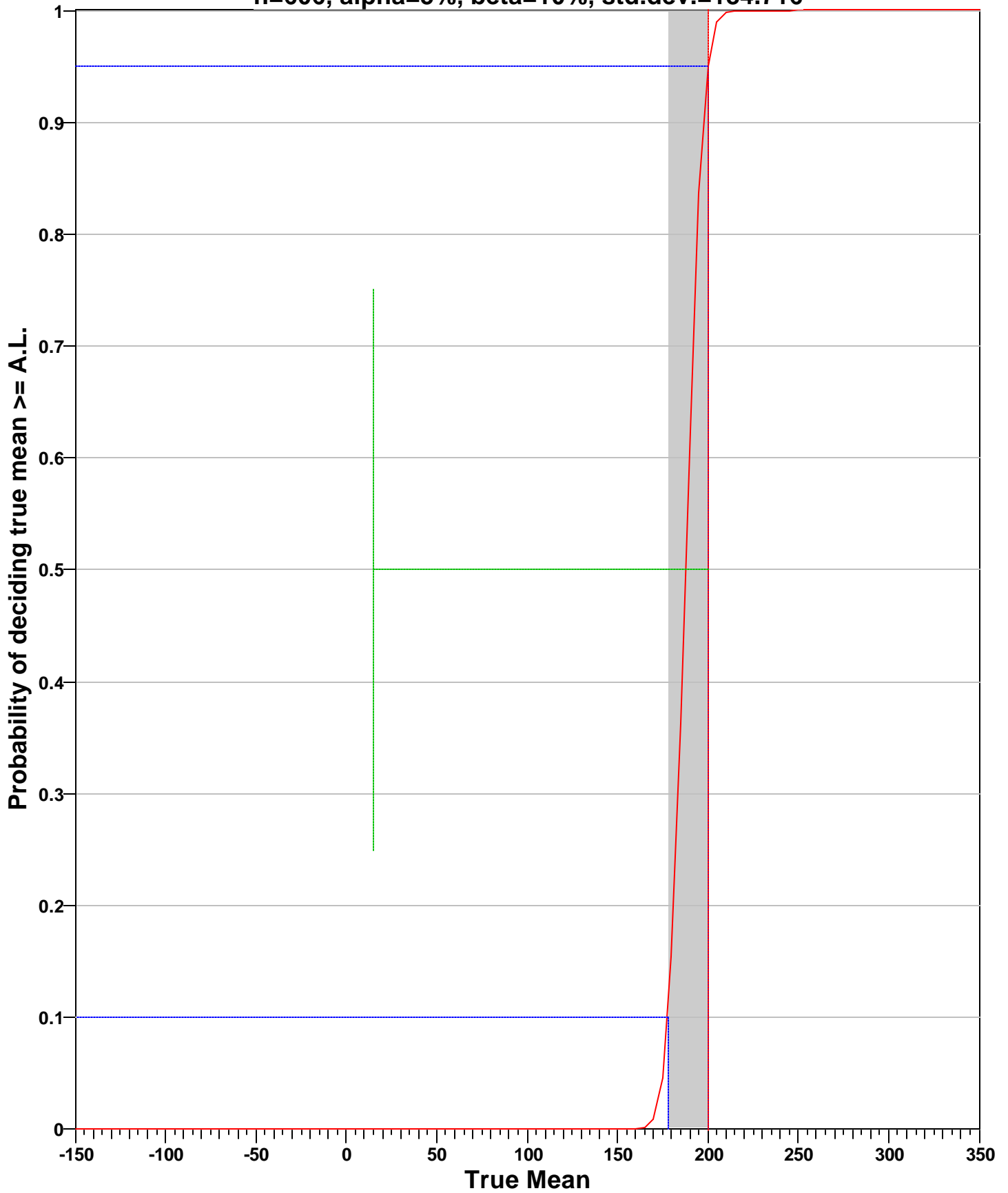
Rockwool DMA
Normal Probability Plot
120 Second Homogenized XRF Samples

Expected Normal Value

XRF Antimony Concentration in mg/kg

Rockwool DMA
Lognormal Probability Plot
120 Second Homogenized XRF Samples

Expected Normal Value

Natural Logarithm of the
XRF Antimony Concentration in mg/kg
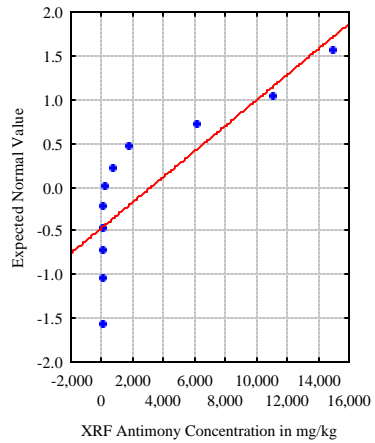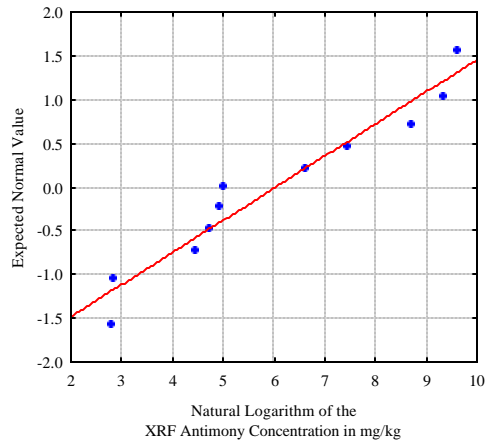
Wilcoxon Signed Rank (One-Sample) Test

n=630, alpha=5%, beta=10%, std.dev.=1.988

Probability of deciding true mean or median >= A.L.

True Mean or Median
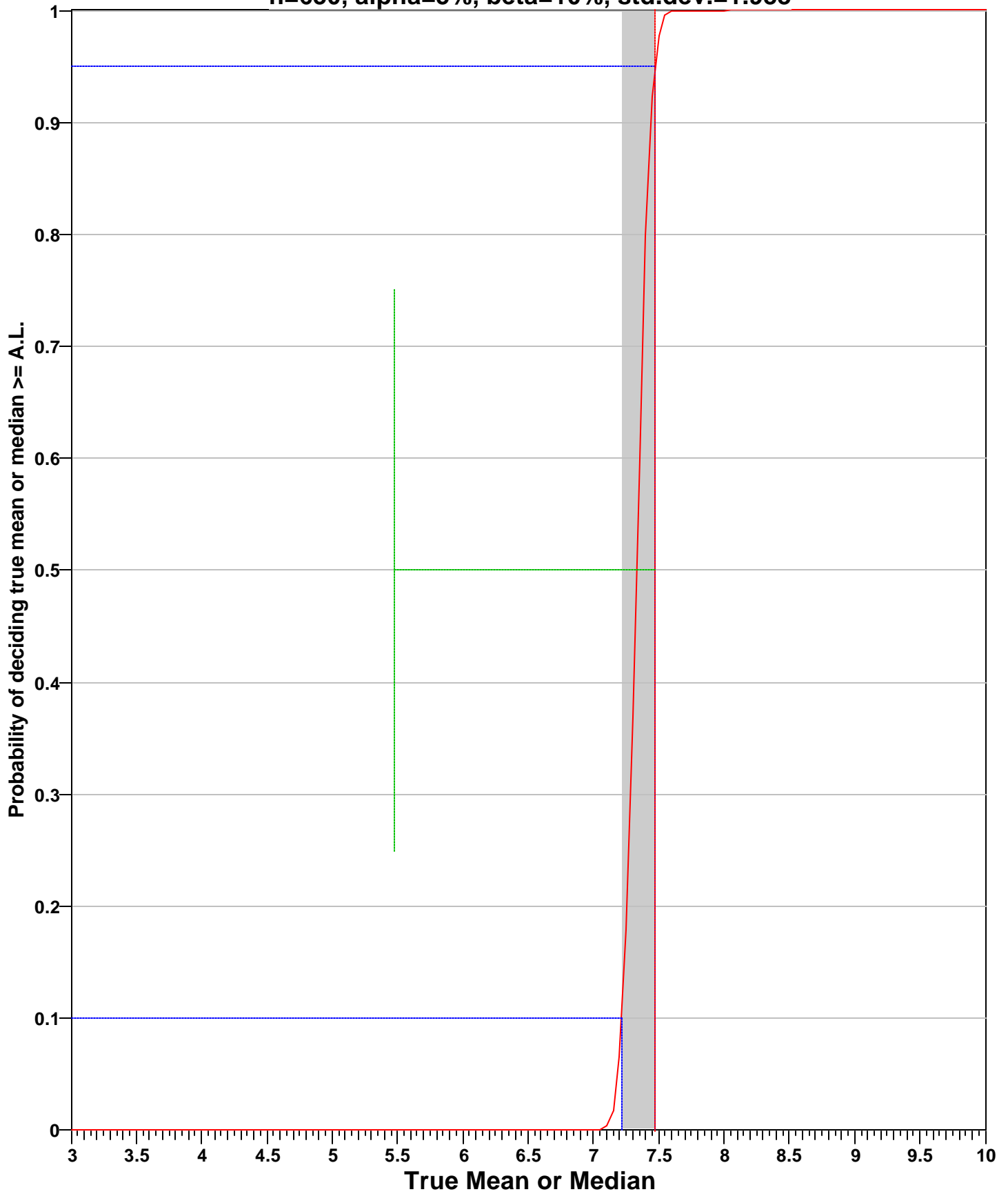
1-Sample t-Test of True Mean vs. Action Level
n=606, alpha=5%, beta=10%, std.dev.=184.716

Probability of deciding true mean >= A.L.

True Mean

Wilcoxon Signed Rank (One-Sample) Test

n=626, alpha=5%, beta=10%, std.dev.=2.457

Probability of deciding true mean or median >= A.L.

True Mean or Median

ATTACHMENT 1

TIER IV LABORATORY DATA